

AD-A271 753



2

ARMY RESEARCH LABORATORY

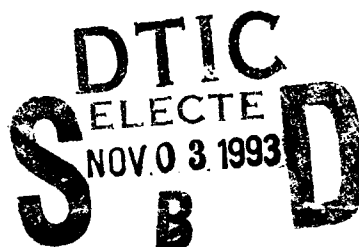


# Stress-Induced Cracking of Fluorocarbon Rubber MTL-0002 After Exposure To DS-2

Karen M. Kinsley

ARL-TR-219

September 1993



93-26479



Approved for public release; distribution unlimited.

93 11 1 070

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

Citation of manufacturer's or trade names does not constitute an official endorsement or approval of the use thereof.

Destroy this report when it is no longer needed. Do not return it to the originator.

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE September 1993		3. REPORT TYPE AND DATES COVERED Final Report
4. TITLE AND SUBTITLE Stress-Induced Cracking of Fluorocarbon Rubber MTL-0002 After Exposure to DS-2			5. FUNDING NUMBERS	
6. AUTHOR(S) Karen M. Kinsley				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Research Laboratory Watertown, MA 02172-0001 ATTN: AMSRL- VS-V			8. PERFORMING ORGANIZATION REPORT NUMBER ARL-TR- 219	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Research Laboratory 2800 Powder Mill Road Adelphi, MD 20783-1145			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words)  Fluorocarbon Rubber 0002 (MTL-0002) (1) was found to be susceptible to stress-induced cracking (SIC) when exposed to the standard decontaminant DS-2 (2). In this report, several tensile tests were conducted upon this rubber and related fluorocarbon elastomers after exposure to DS-2 and its components. It was found that diethylenetriamine, a DS-2 component, was responsible for the formation of cracks under tension. Of the vulcanizates tested, MTL-0002 was the most susceptible to SIC after a one-half hour immersion in DS-2, but I. B. Moore V7D failed at lower stress levels after 24 hours of immersion in DS-2. It is suggested that changing the proportion of magnesium oxide and calcium hydroxide in the recipe and altering the press curing temperature time may enhance the resistance to SIC of MTL-0002.				
14. SUBJECT TERMS Fluorocarbon rubber; Strength of materials			15. NUMBER OF PAGES 21	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL	

## Contents

	Page
Introduction . . . . .	1
Description of MTL-0002 . . . . .	3
Amine and Bisphenol Curing of VF-HFP Fluoroelastomers . . . . .	4
Experimental . . . . .	5
Other Fluoroelastomers . . . . .	9
Analysis . . . . .	11
Conclusions . . . . .	13
Future Work . . . . .	13
Acknowledgments . . . . .	14
References . . . . .	14

## Table

1. MTL-0002 recipe . . . . .	3
------------------------------	---

## Figures

1. Sorption and desorption of MTL-0002 immersed in DS-2 . . . . .	3
2. Photographs of MTL-0002 samples under tension . . . . .	2
3. Average ASTM D 638(V)-87 tensile test results of MTL-00025 after immersion in DS-2 . . . . .	5
4. Structure of bisphenol cured VF/HFP elastomers . . . . .	1
5. Infrared spectrum of control sample of MTL-0002 . . . . .	7
6. Infrared spectrum of MTL-0002 after one hour of immersion in DS-2 . . . . .	7
7. Photograph of MTL-0002 tensile test specimens after tensile testing with varying exposures to droplets of diethylenetriamine . . . . .	8
8. ASTM D 638(V)-87 tensile test results for 40 mil Aflas 0009 samples after DS-2 immersion . . . . .	10
9. ASTM D 638(V)-87 tensile test results for 40 mil I.B. Moore Fluorocarbon Rubber V7D after immersion in DS-2 . . . . .	10
10. ASTM D 638(V)-87 tensile test results of 60 mil Viton Fluoroelastomer after DS-2 immersion . . . . .	11

## Introduction

While conducting a survey of rubber tensile properties after immersion in DS-2 (a mixture of sodium hydroxide (2% by weight), 2-methoxyethanol (28% by weight), and diethylenetriamine (DTA) (70% by weight)), the author noted that while Fluorocarbon Rubber 0002 (MTL-0002) did not sorb more than 2% by weight of DS-2 after 24 hours of immersion (see Figure 1), tensile specimens did undergo stress-induced cracking (SIC) after immersion times of as little as one-half hour (2,3) (see Figures 2 and 3).

Fluorocarbon rubbers are used primarily as seals in harsh environments where the chemical resistance of a fluoroelastomer is required and its expense is justified (4). Surface cracks appearing in such seals while under tension, after the use of DS-2, could pose a hazard for Army personnel and vehicles. The purpose of this paper is to examine the cause of the phenomenon and to describe a possible remedial course of action.

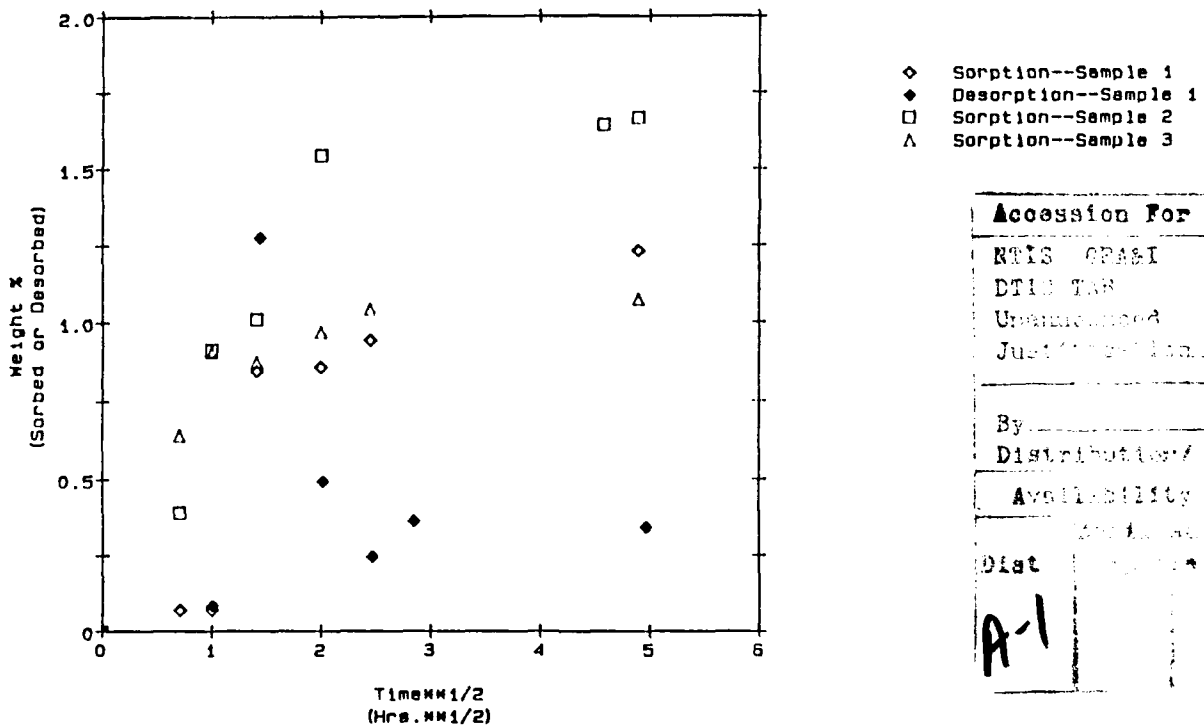
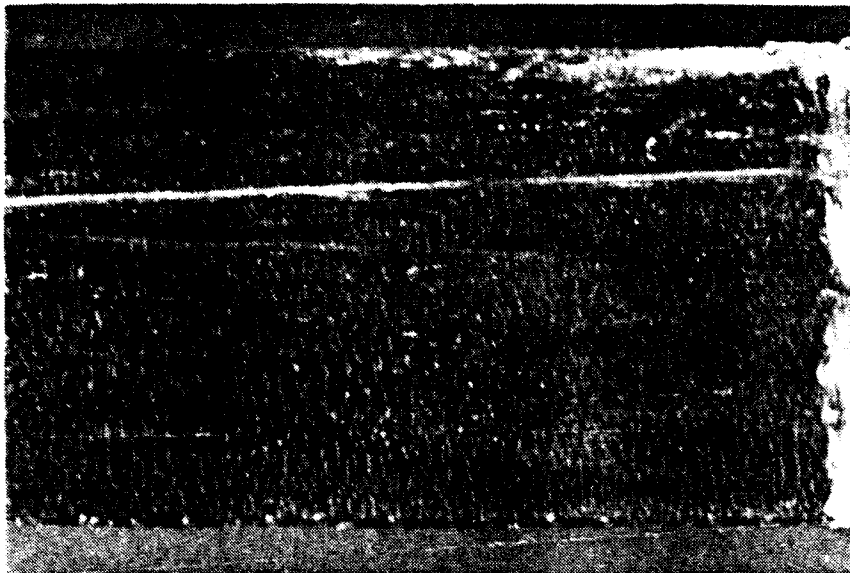
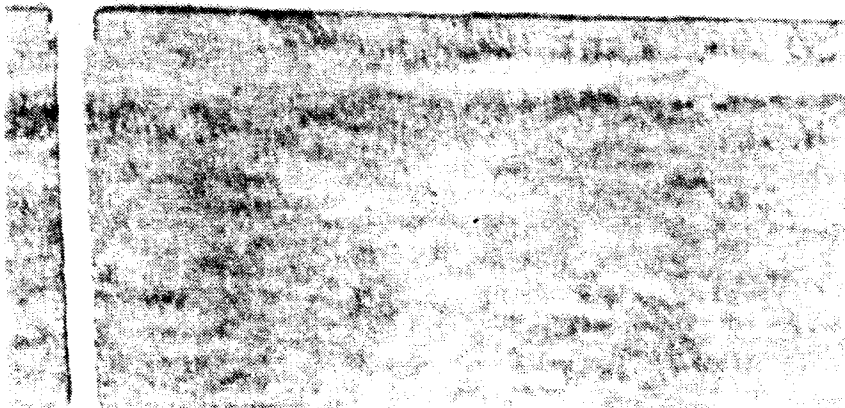


Figure 1. Sorption and desorption of MTL-0002 immersed in DS-2 as taken from Reference 2.



(a)



(b)

Figure 2. Photographs of MTL-0002 samples under tension (taken through telephoto lenses), (a) without prior DS-2 exposure, and (b) after DS-2 immersion, where SIC is visible.

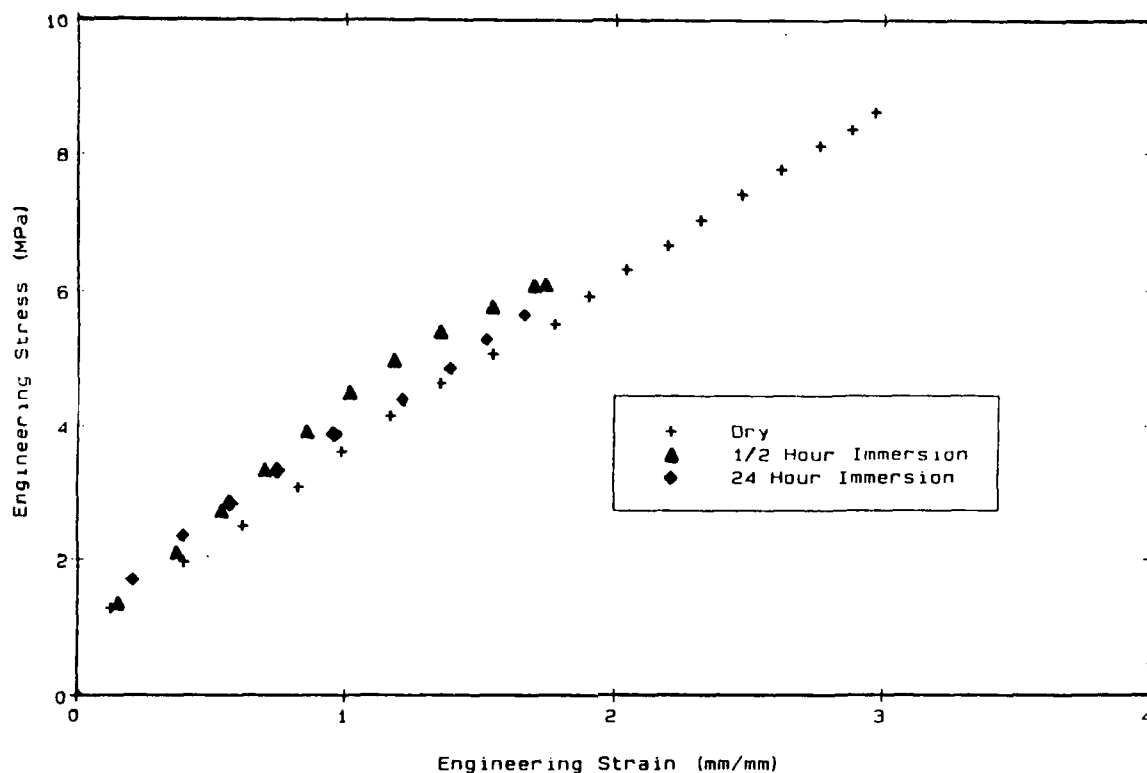


Figure 3. Average ASTM D 638(V)-87 tensile test results of MTL-0002 after immersion in DS-2 as taken from Reference 2.

### Description of MTL-0002

MTL-0002 was prepared by Smithers Scientific Services, Inc. of Akron, OH (1). The recipe is as described in Table 1.

Table 1. MTL-0002 recipe

Component	PHR
Fluorel 2174	80
Fluorel 2145	20
Carnauba Wax	3
N-990 Carbon Black	30
Maglite D (MgO)	6
Calcium Hydroxide	2.5

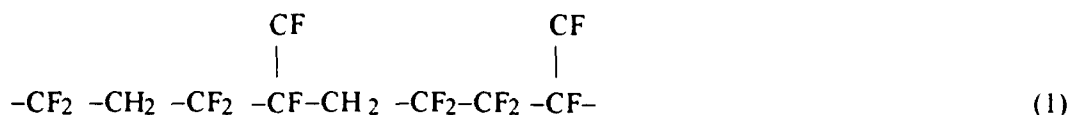
According to Reference 1, the rubber sheets were compression molded at 293°F for 30 minutes. There is no indication of a post-cure heating.

In addition, per Reference 1, the Fluorel gums are copolymers of vinylidene fluoride (VF) and hexafluoropropylene (HFP). Per 3M Corporation (5), the manufacturer of the gums, Fluorel 2145 is supplied without an incorporated curing agent, but can be amine or bisphenol cured. The curing agent is incorporated into Fluorel 2174 (6) and consists of *hexafluoropropylidene diphenol (crosslinker)/triphenylbenzylphosphonium chloride (accelerator)* (7).

The most common general systems employed today for the curing of fluoroelastomers are peroxides, amines, and bisphenols (4,8-13). Peroxide curing of fluoroelastomers is not a factor in SIC of MTL-0002; References 8 and 9 are recommended to the reader interested in this topic.

### Amine and Bisphenol Curing of VF-HFP Fluoroelastomers

The basic structure of VF-HFP fluoroelastomers is (4,8-13):



According to Schmiegel and Logethis (8) and Arcella, et al. (13,14), reactive sites in the presence of the bisphenols are isolated vinylidene fluoride units. In a multistep process, the VF units are dehydrofluorinated (assisted by the action of the onium ion provided by the accelerator) to produce double bonds ( $-\text{C}(\text{CF})=\text{CH}-$ ). The resultant HF then reacts with MgO to produce  $\text{MgF}_2$  and water. (Note: The MgO is required to be present for the dehydrofluorination to occur. Water production competes with the dehydrofluorination and acts as a limiting factor for the reaction (15,17) (also see the Analysis Section of this report)).

Arcella, et al. (13,14) also postulate the formation of  $-\text{CH}=\text{CF}-$  double bonds as an intermediate step. The bisphenol then reacts at the double bond sites to create ether linkages between chains (see Figure 4); HF is again split off from those sites to react with the MgO added during rubber compounding. Calcium hydroxide, added during compounding, is also used as an acid acceptor (1).

Amine curing of VF-HFP fluoroelastomers preceded that of bisphenol or peroxide; the basic research was performed in the late 1950s to early 1960s (4,9-12,15). As with bisphenols, difunctional amines cause dehydrofluorination of VF sites with subsequent formation of carbon-carbon double bonds at those sites. The amines react at the double bond sites to form imine crosslinks and HF, while MgO, added during compounding, reacts with the liberated HF to produce  $\text{MgF}_2$  and water. (This water also acts as a limiting factor on the extent of crosslinking (15).) In addition, tertiary amines were found to cause chain scission of the polymer (10).



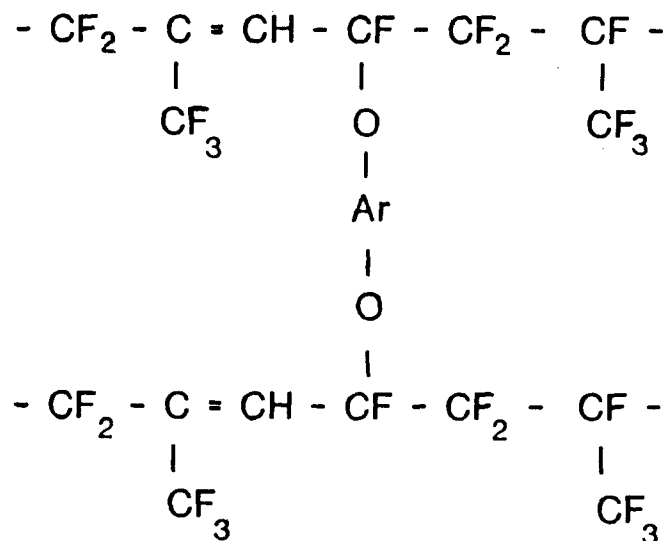


Figure 4. Structure of bisphenol cured VF-HFP elastomers as proposed by Arcella, et al. (see Reference 13).

### Experimental

In the initial study (2), MTL-0002 was cut into tensile specimens per ASTM D 638(V)-87 (16); this size specimen was chosen as a result of limited supplies of Multi-purpose Chemical/Biological Decontaminant (MCBD) microemulsion which was also used in that study. The MTL-0002 samples were immersed for periods of one-half hour or 24 hours in 65 ml of decontaminant, wiped dry with filter paper, and then tested to break in an Instron 1122 tensile test machine with a constant crosshead speed of 50 mm/min. Each series of exposures was conducted separately. Consult Reference 2 for the exact details of the procedure and for the methods used to calculate stress and strain.

As noted in Reference 2, this type of test is not standard for rubber materials and the results obtained should be used for comparison only. For the more normal rubber tensile test, see ASTM D 412-87 (17), and ASTM D 471-79 (18) for immersion of rubber tensile specimens in liquids.

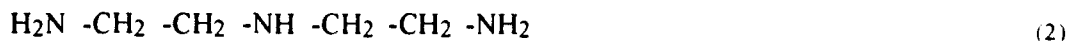
The amount of DS-2 sorbed and desorbed are given in Reference 2 and are displayed in Figure 1. The figure indicates that less than 2% by weight of DS-2 was absorbed by MTL-0002 after 24 hours of immersion. No swelling of MTL-0002 due to DS-2 immersion was noted in Reference 2.

The average ASTM D 638(V)-87 tensile test results for MTL-0002 are reproduced from Reference 2 and are shown in Figure 3. Note the reduction in fracture stress and strain after immersion; this is due to SIC after DS-2 immersion. Photographs of MTL-0002 samples in tension without DS-2 immersion and after immersion (showing SIC) are presented in Figure 2.

In order to determine which components (or combination of components) of DS-2 were responsible for the observed SIC, a series of tests were conducted separately upon sets of two ASTM D 638(V) tensile samples; the results are summarized below:

- After one-half hour immersion in a solution of sodium hydroxide (2% by weight) and 2-methoxyethanol, samples were swollen in a cross-sectional area by an average of 23%. Although multiple surface cracks did not appear, in two cases the samples failed by formation of a single, visible crack at the left edge and progressed across the gage section.
- In contrast, after one-half hour of immersion in pure 2-methoxyethanol no surface cracks formed prior to fracture and samples swelled by an average of 18% in a cross-sectional area.
- SIC was observed in one-half hour of immersion in 2-methoxyethanol (28% by weight) and DTA, and samples were swollen approximately 7% in a cross-sectional area.
- SIC was also seen after immersion in 100% DTA, and appeared in the same manner shown in Figure 2. The samples also felt stiffer after immersion and no swelling occurred.

Diethylenetriamine is a multifunctional amine with the following structure:



From the preceding discussion of amine curing of fluorocarbon gums, it is suggested that the cause of SIC in MTL-0002 after DS-2 immersion is crosslinking at the surface due to the action of DTA.

In order to conform that a chemical reaction did take place at the surface of the samples, three specimens of MTL-0002 were subjected to infrared analysis. One sample was immersed in DS-2, the second immersed in diethylenetriamine while the third was the control. The samples were prepared as follows:

Specimens were cleaned of mold release by rubbing with filter paper, then wiped with ethanol (applied by filter paper) to remove finger oils and were allowed to dry. The specimens immersed in DS-2 and diethylenetriamine were immersed for one hour in their respective liquids; they were separately rinsed in distilled water by immersing for one minute then blotted dry with filter paper.

As can be seen in Figure 5, the control sample exhibits absorption peaks in the OH stretch range ( $3695$  to  $2850 \text{ cm}^{-1}$ ) which is probably due to retained water and other hydroxides generated during curing, the CH range ( $1645$  to  $1290 \text{ cm}^{-1}$ ), and in the CF range ( $1090$  to  $630 \text{ cm}^{-1}$ ) (19,20).

In contrast, the IR spectrum of the DS-2 exposed specimen (see Figure 6) shows reduced presence of OH and CH stretch bands (the peak at  $1425 \text{ cm}^{-1}$  can no longer be seen). The absorption peaks in the  $1400$  to  $1900 \text{ cm}^{-1}$  range are due to water vapor present in the spectrometer. The CF bands exhibit less noise than in the nonimmersed sample, and a new absorption peak (at  $1030 \text{ cm}^{-1}$ ) can be discerned (see Figure 6). The results for DTA immersion are similar to those of DS-2; therefore, the IR data confirm that a surface reaction between the DS-2 and the fluoroelastomer did occur.

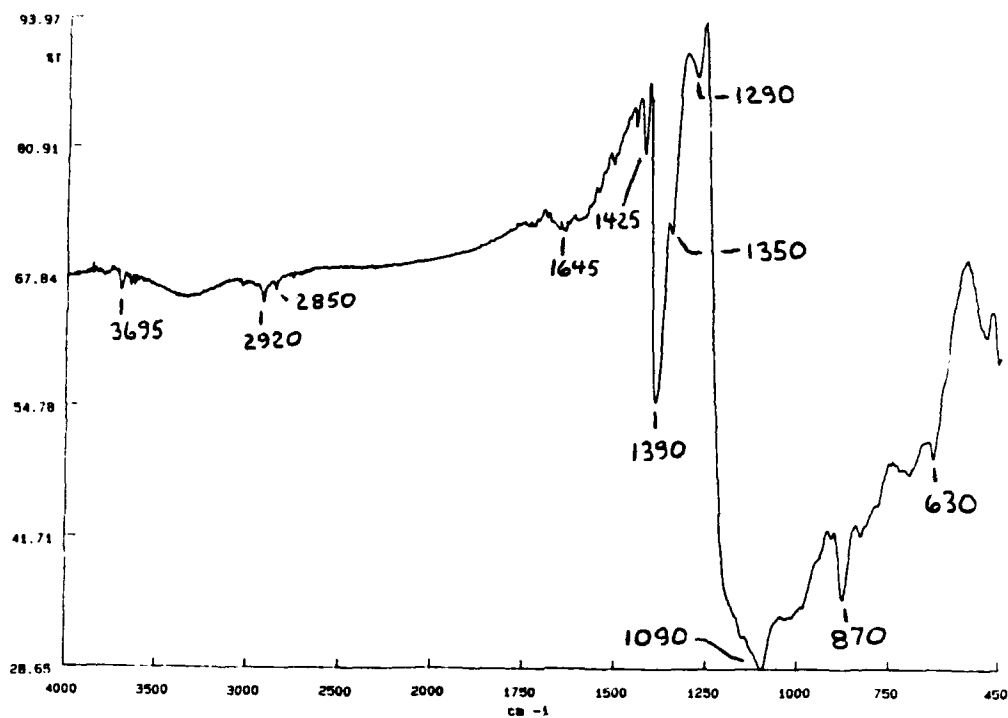


Figure 5. Infrared spectrum of control sample of MTL-0002.

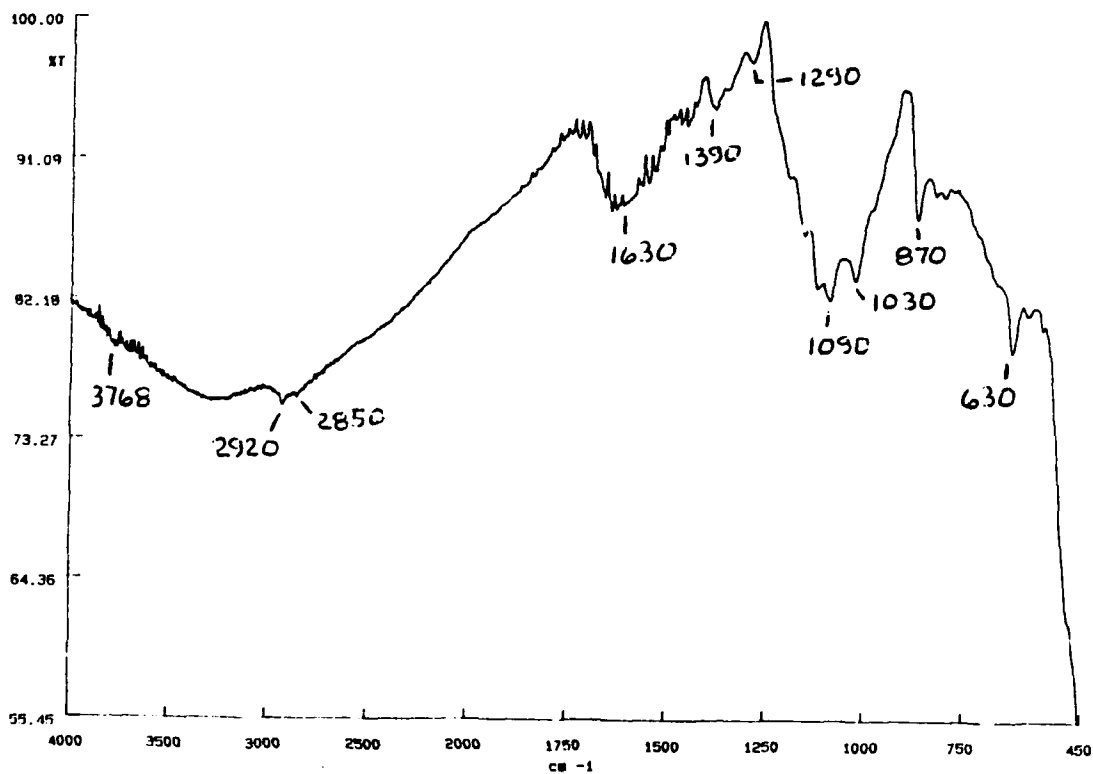


Figure 6. Infrared spectrum of MTL-0002 after one hour of immersion in DS-2.

To determine the approximate time required to cause SIC by DTA, the author placed a single drop of DTA on one side of the gage sections of ASTM D 638(V)-87 samples for various lengths of time; the drops were then wiped off (not rinsed) and the specimens were tested to break.

Figure 7 is a photograph of the results of those tests. With no exposure to DTA (the two samples at the top of the figure), the pattern of fracture is a smooth curve with the center section approximately straight in a  $45^\circ$  angle to the direction of stress (plane of principal stress) indicative of a homogeneous, highly crosslinked material.

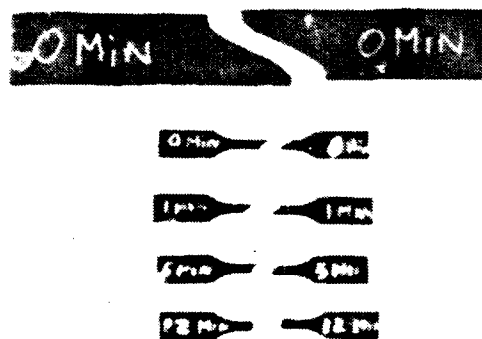


Figure 7 Photograph of MTL-0002 tensile test specimens after tensile testing with varying exposure to droplets of diethylenetriamine. Exposure times were (from the top down): 0 minutes (top two samples), 1 minute, 5 minutes, and 12 minutes.

For an exposure time of one minute, there was no change in the fracture pattern and no observed SIC.

After five minutes there is a change in the fracture pattern: although the major mode of fracture is unchanged, there exists a flat surface (perpendicular to the direction of applied stress) from the fracture plane to the edge. Although no SIC was observed, it is possible that the surface exposed to DTA was subject to the formation of microcracks, and that when the stress was sufficient to fracture the material in bulk, microcracks which had formed perpendicular to the angle of applied stress were sufficient to provide a localized fracture path.

After 12 minutes had elapsed, SIC in the manner of Figure 2 (post DS-2 exposure) was seen and the fracture path was flat and perpendicular to that of the applied stress.

In order to determine the actual elongation of the surface at the first appearance of SIC, MTL-0002 samples were cut into strips and immersed for one-half hour in DS-2 then rinsed in distilled water. These strips were then attached to a second plastic strip (which was approximately 0.1 mm in thickness) via double-sided tape. This assembly was then bent over a conical mandrel, per ASTM D 522-85 (21), and the diameter of the mandrel at which the crack first appeared was recorded. All MTL-0002 specimens were 40 mils in thickness.

ASTM D 522-85 is a test for the flexibility of organic coatings attached to a flexible substrate. This standard determines the percent elongation of the uppermost fibers of the sample (and, hence, the coating) during bending to be:

$$\% \text{Elongation} = \frac{t}{D + t} \cdot (100\%) \quad (3)$$

where:

D = Diameter of the mandrel at which the first crack became visible. (To this was added 2 (thickness of the plastic strip and double-sided tape) in order to obtain the true diameter of the curvature at failure.)

t = Specimen thickness.

This calculation assumes that the substrate and coating deform elastically, that the coating fails in a brittle manner; i.e., without plastic deformation, and that the coating remains bonded to the substrate.

Of seven samples tested, the percent elongation at fracture ranged from 2.46% to 4.75%, with an average value of 3.39% and a standard deviation of  $\pm 0.78\%$ . In contrast, similar tests of unexposed samples showed that cracks appeared only after the strips were folded approximately  $180^\circ$  with a bend radius of 0.70 mm. The calculation method given above yields a percent elongation of 60.5% for these samples.

### Other Fluoroelastomers

To determine if SIC is a problem only with MTL-0002, or whether this was important to other Army fluorocarbon elastomers, two sets of two ASTM D 638-87 (Type V) samples of Aflas-0009 (1), I.B. Moore Fluorocarbon Rubber V7D (1), and Viton (1) (exact composition unknown) were immersed for one-half hour and 24 hours in DS-2 and then subjected to tensile testing as described above, except that the 24 hour specimens were rinsed for one minute in distilled water.

The results are displayed in Figures 8 through 10. Aflas is a copolymer of tetrafluoroethylene and propylene (1,22,23) and is, therefore, highly resistant to most chemicals. Although some stiffening of the samples are noted after 24 hours exposure, no SIC was observed.

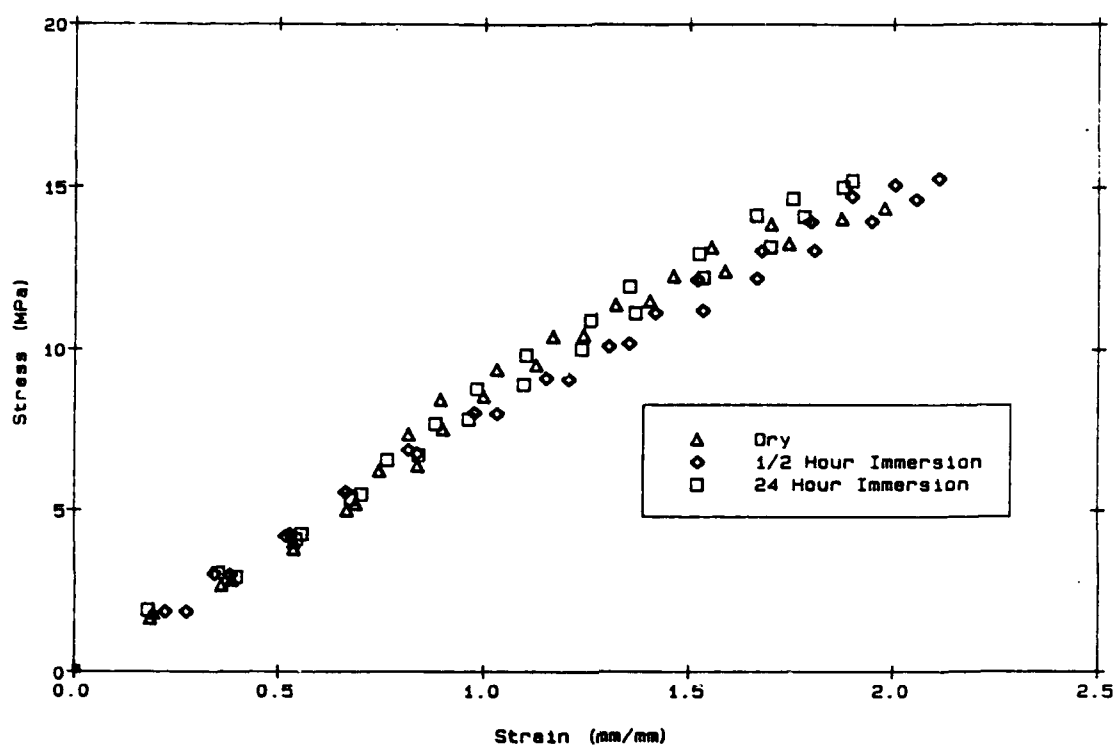


Figure 8. ASTM D 638(V)-87 tensile test results for 40 mil Aflas 0009 samples after DS-2 immersion.

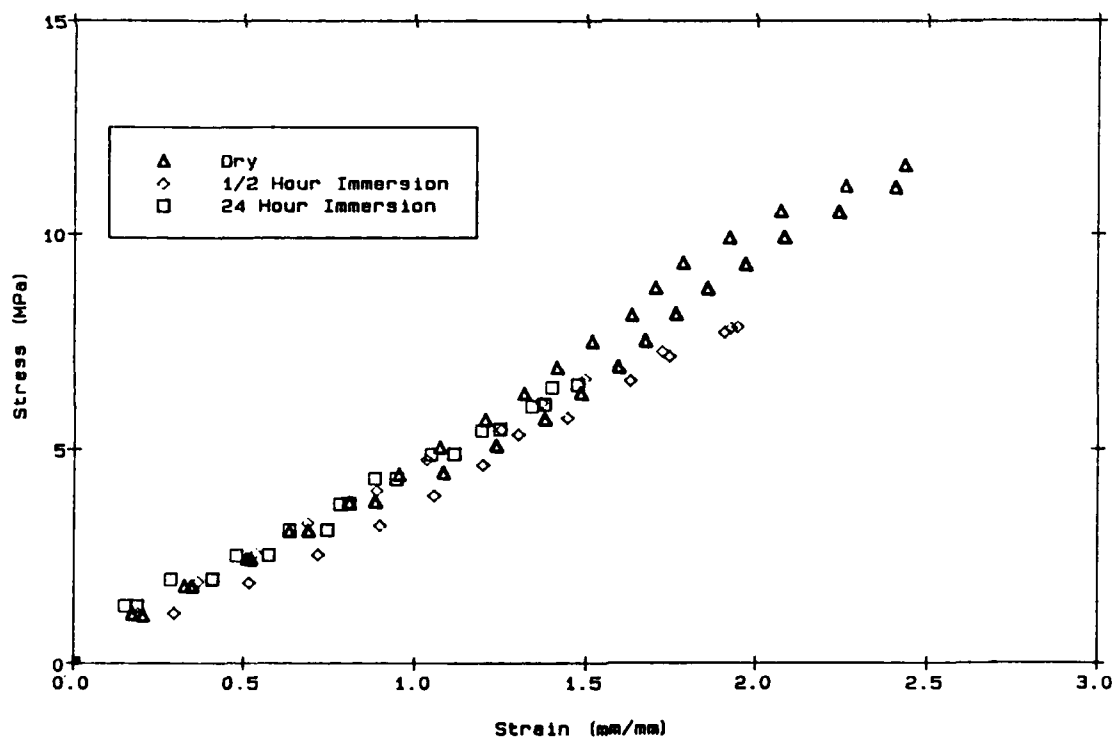


Figure 9. ASTM D 638(V)-87 tensile test results for 40 mil I.B. Moore Fluorocarbon Rubber V7D after immersion in DS-2

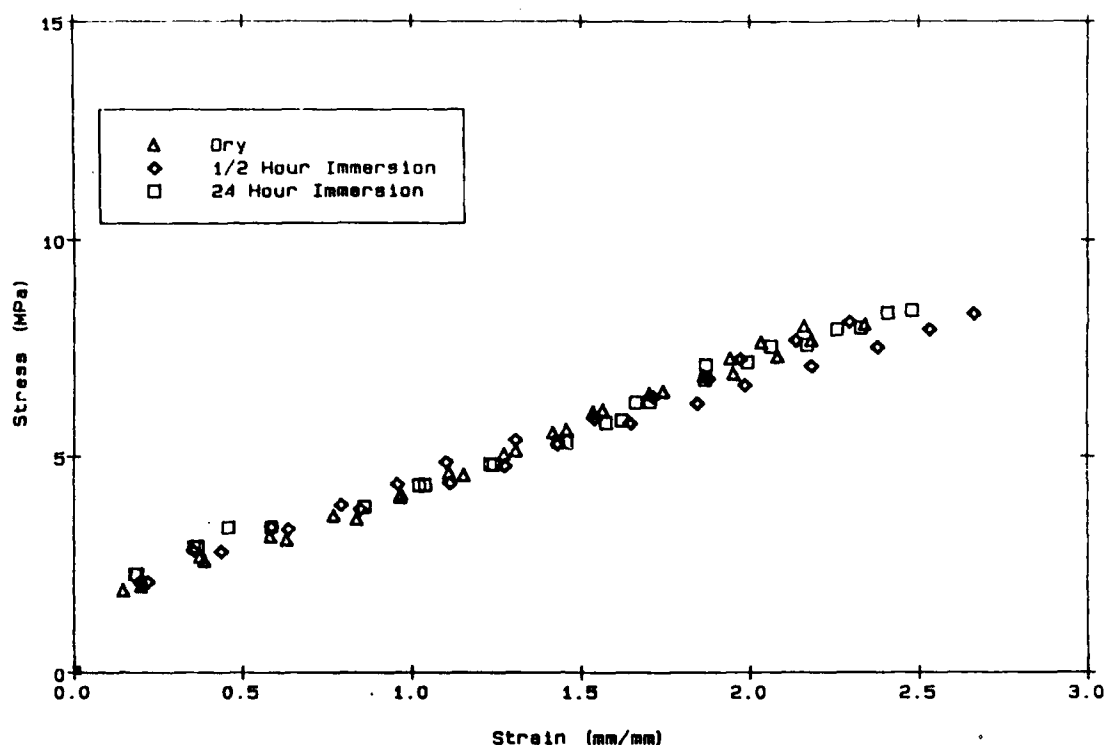


Figure 10. ASTM D 638(V)-87 tensile test results of 60 mil Viton Fluoroelastomer after DS-2 immersion.

Both the Viton (4,23) and I.B. Moore fluoroelastomers (1) are copolymers of vinylidene fluoride and hexafluoropropylene as is MTL-0002. For both of these fluoroelastomers, the SIC was observed prior to fracture after one-half hour of immersion; the I.B. Moore fluoroelastomer failed more quickly than the Viton, due in part to the differences in thickness (40 mils for V7D versus 60 mils for the Viton) and the resultant resistance to cracking given by the greater thickness. The I.B. Moore vulcanizate also failed at a higher stress than MTL-0002 after one-half hour of immersion, but at a low stress after 24 hours of exposure.

### Analysis

It is evident that the VF-HFP elastomers tested are susceptible to SIC after DS-2 exposure. This attack appears to be due to diethylenetriamine acting as a crosslinker on the fluorocarbon rubber surfaces, possibly aided by the presence of NaOH acting as an acid acceptor (studies of the effects of aqueous solutions of NaOH on poly(vinylidene fluoride) (24,25) at temperatures between 20°C through 100°C and at concentrations between 5% through 50% have shown that sodium hydroxide at elevated temperatures and concentrations will dehydrofluorinate poly(vinylidene fluoride), both as a single solute (24) and in the presence of quaternary ammonium or phosphonium halides (25).

This crosslinking is a surface phenomenon and is supported by the low sorption of DS-2 by MTL-0002 (see Figure 1), the high stress level at sample fracture despite the presence of SIC, and by the lack of swelling in the presence of 2-methoxyethanol in the full DS-2 solution only. (Swelling of FKM vulcanizates in the presence of 2-methoxyethanol (methyl cellosolve) is noted in the literature (26-28).)

Comparison of the tensile results (see Figures 3 and 8 through 10) indicates that MTL-0002 and Fluorocarbon Rubber V7D are the most vulnerable to SIC of the elastomers tested. The recipes (1) for these rubbers are similar; the main difference is in the proportion of MgO and calcium hydroxide used: MTL-0002 used 6 phr MgO and 2.5 phr Ca(OH)<sub>2</sub> while V7D used 3 phr MgO and 6 phr Ca(OH)<sub>2</sub>.

The author corresponded with 3M Corporation (29), the manufacturer of the Fluorel gums, regarding the recipe for MTL-0002. 3M indicated that there was sufficient curative present in Fluorel 2174 to allow proper cure of the Fluorel 2145 also present in the recipe. It was indicated that *optimum physical properties are obtained by a combination of 3 parts Maglite D (MgO) and 6 parts calcium hydroxide* (note that this is the V7D proportioning) and that better compression set values could be obtained by using 1 to 2 phr Carnauba Wax instead of the 3 phr employed.

3M also recommended changing the cure time and temperature of MTL-0002 from 30 minutes at 293°F and 10 minutes at 325°F to 350°F, and then to *post-cure at 450°F for approximately 16 hours*. No post-cure was specified for MTL-0002 (see References 4 through 7).

It is possible that a greater degree of cure could be obtained at the higher temperature, and it is suggested that if this is the case, it is possible that the vulnerability to SIC in MTL-0002 after DS-2 exposure could be reduced.

However, the recommendation regarding post-cure should not be followed. Although Paciorek (12) showed that heating after press curing does induce further crosslinking in the rubber, and Smith and Perkins (15) linked this behavior to the elimination of water (known to act as a control on the degree of crosslinking in *cis*-vulcanizate), recent studies (see below) have indicated that this additional crosslinking is not stable over time.

Ferro, et al. (30) have studied the post-cure behavior of bisphenol cured FKM rubbers and have concluded that mechanical properties developed during the post-cure heating degrade in normal room storage conditions because the majority of bonds created during post-cure are physical in nature (formed between the filler and the elastomer matrix because of the elimination of water during heating); they are broken due to the re-sorption of water from the atmosphere. It was found that the most stable properties were achieved by those materials which were only press-cured. Samples which were stored at 50% relative humidity required a second post-cure to restore maximum strength to the elastomers.

These results were confirmed by Struckmeyer (31), who also noted that this phenomenon of "drifting" occurred in both bisphenol and peroxide cured vulcanizates, although the behavior changes due to post-cure water sorption varied according to the curing system employed.

References 30 and 31 noted that rubbers filled with nonblack, hydrophobic fillers were the most stable over time and that while carbon black filled vulcanizates were more stable than most other nonblack fillers, they were nevertheless subject to "drifting".

Fluorocarbon Rubber 0002 was compounded in order to generate consistent results for many types of tests and test conditions and to, therefore, provide a reference for testing



other fluoroelastomers. If a post-cure heat treatment were given to the newly formed sheets, the degree of "drift" from the initial tensile data would have to be evaluated; i.e., "calibration" would be required, each time a sheet is used. If the initial mechanical properties were desired, then a second heat treatment may well have to be employed. This may not often be feasible, especially if the sheet(s) were to be employed in field testing or for contractor use where controlled storage conditions and/or curing ovens may be present. Therefore, in order to maximize the stability of mechanical properties of MTL-0002 over time, it is recommended that only a press cure be applied, albeit at the greater temperatures recommended by 3M Corporation (29) as noted above.

## Conclusions

The fluorocarbon elastomers based upon poly(vinylidene fluoride-co-hexafluoropropylene) tested in this study (MTL-0002, I.B. Moore V7B, and a Viton elastomer) are susceptible to SIC after one-half hour immersion in DS-2, while Aflas 0009 (based upon poly(tetrafluorethylene-copolypropylene)) is not so affected. The cause of this vulnerability appears to be crosslinking on the surfaces of the polymers induced by diethylenetriamine, one of the components of DS-2.

In order to increase resistance to this attack, changes in the recipe of MTL-0002 to increase the degree of crosslinking should be employed; in correspondence with 3M Corporation (29), these should include changing the press cure temperature and altering the levels of Maglite D (MgO) and calcium hydroxide. If additional compression set resistance is desired, the level of Carnuba Wax should also be reduced.

However, a post-cure heat treatment is not recommended; Ferro, et al. (30) and Struckmeyer (31) have found that the additional strength imparted to FKM vulcanizates which have been subjected to a post-cure heat treatment have been found to be unstable over time because the bonds so created are primarily physical in nature and are formed by the desorption of water. Re-sorption of water from the atmosphere during storage reduces the strength of the rubber. The initial properties imparted by the press cure do appear to be stable in storage. It is, therefore, recommended that the application of a post-cure heat treatment to MTL-0002 not be employed.

## Future Work

Due to the presence of amine additives in the new SG grade automobile engine oils (13,32,33), and the increasing use of "gasohol" fuels (gasoline and alcohol) (34), the rubber industry has recently become concerned that hoses and seals in automobile engine environments may fail prematurely. New base-resistant fluorelastomers have been patented; for example, see References 35 and 36, in response to this concern. Future work should include obtaining samples of these new materials (along with others which may have been patented since the writing of this report) and testing them for resistance to DS-2 and other decontaminants, chemical/biological warfare agents and stimulants, and for petroleum/oil/lubricant resistance.

## Acknowledgments

The author would like to thank Anthony Wilde, Robert Singler, and Wenzel Davidsohn for their support during this project; Thomas Kenney and Scott Clark for performing the IR; and James Sloan for interpreting the results. The author would also like to especially thank Domenic Macaione for his time and talents in taking and developing the photographs used in this report.

## References

1. WILDE, A. F. *Standard Rubbers for Chemical Defense Research, Volume I: Formulation and Properties*. MTL TR 89-5, U.S. Army Materials Technology Laboratory, January 1989.
2. KINSLEY, K. M. *The Effects of MCBD Microemulsion and DS-2 Upon the Tensile Properties of Various Elastomers*. MTL TR 92-70, U.S. Army Materials Technology Laboratory, September 1992.
3. WILDE, A. F., KINSLEY, K. M., and SEVERE, G. *The Compatibility of Three Decontaminants with Polymeric Materials, Status Report*. To Be Issued as a Technical Report. U.S. Army Materials Technology Laboratory, September 1990.
4. *Encyclopedia of Polymer Science and Engineering*, Volume Seven. John Wiley and Sons, New York, NY, 1987, p. 257269.
5. Fluorel FC Fluoroelastomer 2145. 3M Report No. Y-IFE2145. Commercial Chemicals Division/3M, 3M Corporation, St. Paul, MN, June 1984.
6. Fluorel FC Fluoroelastomer 2174. 3M Report No. Y-IFE2174. Commercial Chemicals Division/3M, 3M Corporation, St. Paul, MN, June 1984.
7. *Fluorel Fluoroelastomer Compounding Information*. 3M Corporation Technical Report No. 98-021-2127-6. Commercial Chemicals Division/3M, 3M Corporation, St. Paul, MN. Not Dated.
8. SCHMIEGEL, W. W., and LOGETHIS, A. L. *Curing of Vinylidene Fluoride Based Elastomers*. American Chemical Society (ACS) Symposium Series No. 260. American Chemical Society, Washington, DC, 1984, p. 159-182.
9. OGUNNIYI, D. S. *The Curing of Fluoroelastomers*. *Progress in Rubber and Plastics Technology*, v. 5, no. 1, 1989, p. 16-25.
10. SMITH, J. F. *The Chemistry of "Viton" A Fluorocarbon Elastomer*. *Rubber World*, v. 142, June 1960, p. 102-107.
11. PACIOREK, K. L., MITCHELL, L. C., and LENK, C. T. *Mechanism of Amine Crosslinking of Fluoroelastomers. I. Solution Studies*. *Journal of Polymer Science*, v. XLV, 1960, p. 405-413.
12. PACIOREK, K. L., LAGINESS, W. G., and LENK, C. T. *Differential Thermal Analysis of Fluoroelastomer Systems*. *Journal of Polymer Science*, v. 60, 1962, p. 141-148.
13. ARCELLA, V., ALBANO, M., BARCIGESI, E., BRINATI, G., and CHIODINI, G. *Development of New Nucleophile Resistant Vinylidene Fluoride Fluorocarbon Elastomers*. *Rubber World*, v. 207, March 1993, p. 18-22.
14. ARCELLA, V., CHIODINI, G., DeFANTI, N., and PIANCA, M. *Crosslinking Chemistry of VF2 Fluorocarbon Elastomers by Bis-Nucleophiles*. Paper No. 57, Presented at the American Chemical Society Rubber Division Meeting, Detroit, MI, October 8-11, 1991.
15. SMITH, J. F., and PERKINS, G. T. *The Mechanism of Post Cure of Viton A Fluorocarbon Elastomer*. *Journal of Applied Polymer Science*, v. V, no. 16, 1961, p. 460-467.
16. *Standard Test Method for Tensile Properties of Plastics*. ASTM D 638-87.
17. *Standard Test Method for Tensile Properties of Rubbers*. ASTM D 412-87.
18. *Standard Test Method for Rubber Property - Effect of Liquids*. ASTM D 471-79.
19. *The Infrared Spectra Atlas of Monomers and Polymers*. Sadtler Research Laboratories, Division of Bio-Rad Laboratories, Incorporated, Philadelphia, PA, 1980, p. 165-172.
20. MORRISON, R., and BOYD, R. *Organic Chemistry, Fourth Edition*. Allyn and Bacon, Inc., Boston, MA, 1983, p. 682.

21. *Standard Test Method for Elongation of Attached Organic Coatings with Conical Mandrel.* ASTM D 522-85.
22. *Aflas TFE Elastomer.* 3M Corporation Technical Report No. 98-0211-4610-9 (69.2 R1), 3M Industrial Chemical Products Division, 3M Corporation, St. Paul, MN, June 1989.
23. BYRNE, C. A. *Studies of Aflas Fluoroelastomers.* MTL TR 89-69, U.S. Army Materials Technology Laboratory, July 1989.
24. BARTH, E. *Verhalten von Polyvinylidenfluorid (PVDF) gegen Natronlauge.* *Kunststoffe*, v. 72, 1982, p. 300-302.
25. KISE, H., and OGATA, H. *Phase Transfer Catalysis in Dehydrofluorination of Poly(vinylidenefluoride) by Aqueous Sodium Hydroxide Solutions.* *Journal of Polymer Science, Polymer Chemistry*, ed., v. 21, 1983, p. 3443-3451.
26. *Fluid Sealing Association Technical Handbook, Properties and Chemical Compatibility of Elastomers for Seals.* Fluid Sealing Association, Philadelphia, PA, 1978.
27. PRUETT, K. M. *Chemical Resistance Guide for Elastomers.* Compass Publications, La Mesa, CA, 1988.
28. *The General Chemical Resistance of Various Elastomers.* 1970 Yearbook of the Rubber Group, Inc., Los Angeles, CA, 1970.
29. Private Letter from Ted Zymczak to Karen M. Kinsley regarding the recipe used in MTL-0002.
30. FERRO, R., GIUNCHI, G., ALOYSIO, S., and LAGNA, C. *Effect of Environmental Conditions, Fillers on Cured Elastomers.* *Rubber and Plastics News 1990 Technical Notebook*, March 1991, p. 12-18.
31. STRUCKMEYER, H. F. *Drifting of Fluorocarbon Elastomer Vulcanizates as a Function of Water Interaction.* *Journal of Elastomers and Plastics*, v. 24, January 1992, p. 55-83.
32. GROOTAERT, W. M., KOLB, R. E., and WORM, A. T. *A Novel Fluorocarbon Elastomer for High Temperature Sealing Applications in Aggressive Motor Oil Environments.* *Rubber Chemistry and Technology*, v. 63, 1992, p. 516-522.
33. ARCELLA, V., FERRO, R., ALBANO, M., and MINUTILLO, A. *New Peroxide Curable Fluoroelastomers with Improved Mechanical Properties and Processability.* *Kautschuk Gummi Kunststoffe*, v. 44, September 1991, p. 833-837.
34. MATROMATTEO, R. *Elastomer Use in Alternate Fuels.* *Rubber and Plastics News 1990 Technical Yearbook*, March 1991, p. 84-87.
35. *Base Resistant Fluoroelastomers with Improved Processability and Curability.* U. S. Patent No. 5,102, 965, Issued April 7, 1992.
36. *Curable Mixes of Fluoroelastomers Containing Bromine or Iodine and of Organic Peroxides.* U.S. Patent No. 5, 153, 272, Issued October 6, 1992.

# DISTRIBUTION LIST

No. of Copies	To
1	Office of the Under Secretary of Defense for Research and Engineering, The Pentagon, Washington, DC 20301
	Director, U.S. Army Research Laboratory, 2800 Powder Mill Road, Adelphi, MD 20783-1197
1	ATTN: AMSRL-OP-CI-AD, Technical Publishing Branch
1	AMSRL-OP-CI-AD, Records Management Administrator
	Commander, Defense Technical Information Center, Cameron Station, Building 5, 5010 Duke Street, Alexandria, VA 23304-6145
2	ATTN: DTIC-FDAC
1	MIA/CINDAS, Purdue University, 2595 Yeager Road, West Lafayette, IN 47905
	Commander, Army Research Office, P.O. Box 12211, Research Triangle Park, NC 27709-2211
1	ATTN: Information Processing Office
	Commander, U.S. Army Materiel Command, 5001 Eisenhower Avenue, Alexandria, VA 22333
1	ATTN: AMCSCI
	Commander, U.S. Army Materiel Systems Analysis Activity, Aberdeen Proving Ground, MD 21005
1	ATTN: AMXSY-MP, H. Cohen
	Commander, U.S. Army Missile Command, Redstone Arsenal, AL 35809
1	ATTN: AMSMI-RD-CS-R/Doc
	Commander, U.S. Army Armament, Munitions and Chemical Command, Dover, NJ 07801
2	ATTN: Technical Library
	Commander, U.S. Army Natick Research, Development and Engineering Center, Natick, MA 01760-5010
1	ATTN: Technical Library
	Commander, U.S. Army Satellite Communications Agency, Fort Monmouth, NJ 07703
1	ATTN: Technical Document Center
	Commander, U.S. Army Tank-Automotive Command, Warren, MI 48397-5000
1	ATTN: AMSTA-ZSK
1	AMSTA-TSL, Technical Library
	Commander, White Sands Missile Range, NM 88002
1	ATTN: STEWS-WS-VT
	President, Airborne, Electronics and Special Warfare Board, Fort Bragg, NC 28307
1	ATTN: Library
	Director, U.S. Army Research Laboratory, Weapons Technology, Aberdeen Proving Ground, MD 21005-5066
1	ATTN: AMSRL-WT

No. of Copies	To
1	Commander, Dugway Proving Ground, UT 84022 ATTN: Technical Library, Technical Information Division
1	Commander, U.S. Army Research Laboratory, 2800 Powder Mill Road, Adelphi, MD 20783 ATTN: AMSRL-SS
1	Director, Benet Weapons Laboratory, LCWSL, USA AMCCOM, Watervliet, NY 12189 ATTN: AMSMC-LCB-TL
1	AMSMC-LCB-R
1	AMSMC-LCB-RM
1	AMSMC-LCB-RP
3	Commander, U.S. Army Foreign Science and Technology Center, 220 7th Street, N.E., Charlottesville, VA 22901-5396 ATTN: AIFRTC, Applied Technologies Branch, Gerald Schlesinger
1	Commander, U.S. Army Aeromedical Research Unit, P.O. Box 577, Fort Rucker, AL 36360 ATTN: Technical Library
1	U.S. Army Aviation Training Library, Fort Rucker, AL 36360 ATTN: Building 5906-5907
1	Commander, U.S. Army Agency for Aviation Safety, Fort Rucker, AL 36362 ATTN: Technical Library
1	Commander, Clarke Engineer School Library, 3202 Nebraska Ave., N, Fort Leonard Wood, MO 65473-5000 ATTN: Library
1	Commander, U.S. Army Engineer Waterways Experiment Station, P.O. Box 631, Vicksburg, MS 39180 ATTN: Research Center Library
1	Commandant, U.S. Army Quartermaster School, Fort Lee, VA 23801 ATTN: Quartermaster School Library
2	Naval Research Laboratory, Washington, DC 20375 ATTN: Dr. G. R. Yoder - Code 6384
1	Chief of Naval Research, Arlington, VA 22217 ATTN: Code 471
1	Commander, U.S. Air Force Wright Research & Development Center, Wright-Patterson Air Force Base, OH 45433-6523 ATTN: WRDC/MLLP, M. Forney, Jr.
1	WRDC/MLBC, Mr. Stanley Schulman
1	U.S. Department of Commerce, National Institute of Standards and Technology, Gaithersburg, MD 20899 ATTN: Stephen M. Hsu, Chief, Ceramics Division, Institute for Materials Science and Engineering

No. of Copies	To
1	Committee on Marine Structures, Marine Board, National Research Council, 2101 Constitution Avenue, N.W., Washington, DC 20418
1	Materials Sciences Corporation, Suite 250, 500 Office Center Drive, Fort Washington, PA 19034
1	Charles Stark Draper Laboratory, 555 Technology Square, Cambridge, MA 02139
	Wyman-Gordon Company, Worcester, MA 01601
1	ATTN: Technical Library
	General Dynamics, Convair Aerospace Division, P.O. Box 748, Fort Worth, TX 76101
1	ATTN: Mfg. Engineering Technical Library
	Plastics Technical Evaluation Center, PLASTEC, ARDEC, Bldg. 355N, Picatinny Arsenal, NJ 07806-5000
1	ATTN: Harry Pebly
1	Department of the Army, Aerostructures Directorate, MS-266, U.S. Army Aviation R&T Activity - AVSCOM, Langley Research Center, Hampton, VA 23665-5225
1	NASA - Langley Research Center, Hampton, VA 23665-5225
	U.S. Army Vehicle Propulsion Directorate, NASA Lewis Research Center, 2100 Brookpark Road, Cleveland, OH 44135-3191
1	ATTN: AMSRL-VP
	Director, Defense Intelligence Agency, Washington, DC 20340-6053
1	ATTN: ODT-5A (Mr. Frank Jaeger)
	U.S. Army Communications and Electronics Command, Fort Monmouth, NJ 07703
1	ATTN: Technical Library
	U.S. Army Research Laboratory, Electronic Power Sources Directorate, Fort Monmouth, NJ 07703
1	ATTN: Technical Library
	Director, U.S. Army Research Laboratory, Watertown, MA 02172-0001
2	ATTN: AMSRL-OP-CI-D, Technical Library
5	Author